

VISIT...

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REFLUX: MICROSCALE

There's not much difference between the microscale reflux and the bigger ones other than the size of the glassware, so much of the information for the larger setup will still apply (see Chapter 22, "Reflux"). And if you want a dry microscale reflux, then add a drying tube (Fig. 113).

ADDITION AND REFLUX: MICROSCALE

Once you get used to the idea of handling reagents in syringes (see Chapter 8, "Syringes and Needles"), this is pretty straightforward (Fig. 114). Sometimes you have to watch out for back-pressure in the setup, and you have to hold the syringe plunger a bit to keep it from blowing back out. Normally, you just hold the barrel of the loaded syringe, pierce the septum, and then add the reagent, a little at a time, into the setup. How fast? Usually as fast as practical. If the reaction speeds up too much, the ring of condensate in the reflux condenser will travel up and out of the condenser and into the room air. No, no, no, no. This sounds identical to the warning I give for the large-scale setup. **Hint:** It is.

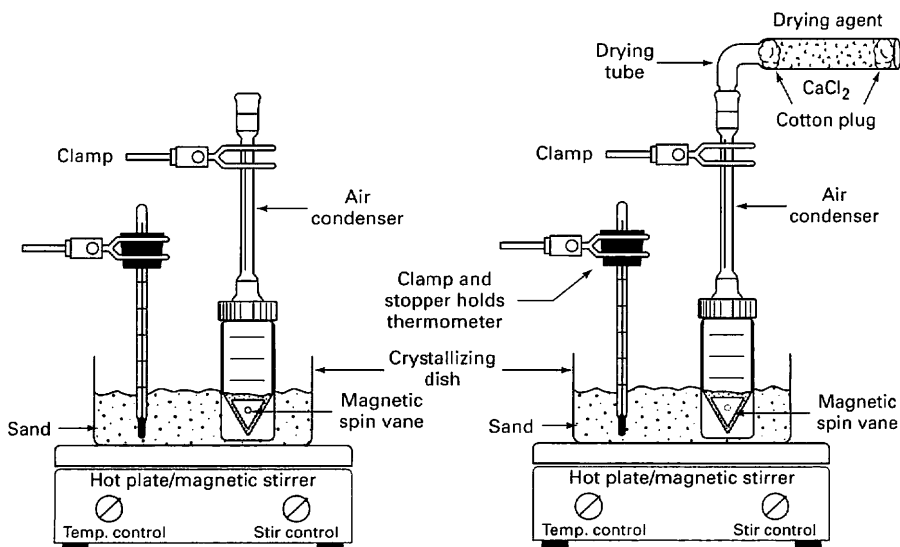


Fig. 113 Microscale reflux both wet and dry.

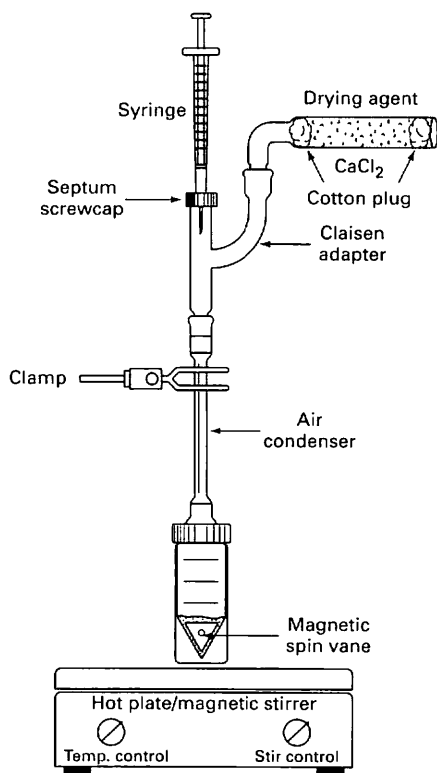


Fig. 114 A dry microscale reflux and addition.